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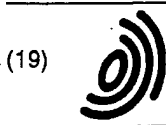
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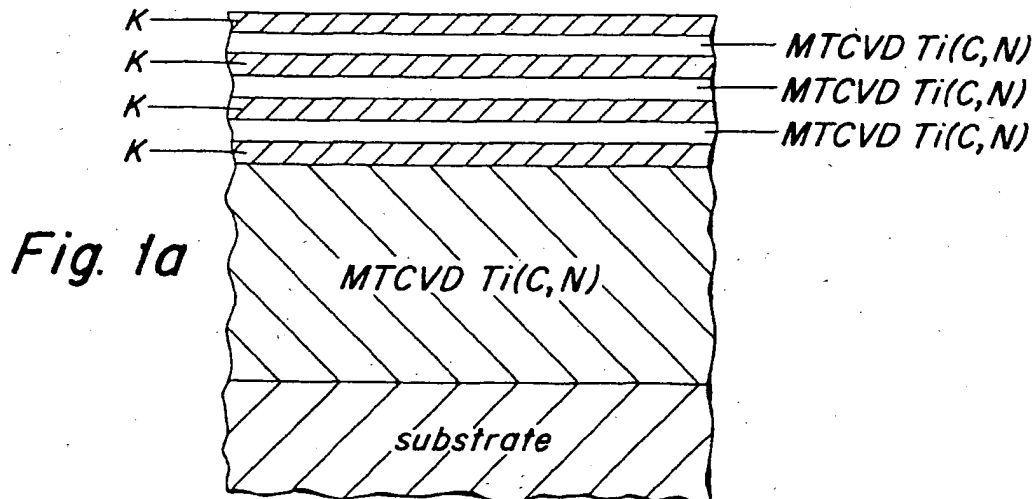
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(54) Kappa and/or gamma A1203 multi-coating deposited by low temperature CVD

(57) A coated body having a multi-layer of  $\kappa$ -Al<sub>2</sub>O<sub>3</sub> and or  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> or TiN applied by MTCVD (Medium Temperature Chemical Vapor Deposition) is disclosed. The multi-layers can be interspersed with layers of Ti(C,N)

which can also be applied by MTCVD. The body which is coated is preferably a cemented carbide, cermet, ceramic and/or high speed steel and may be used as a metal cutting insert.



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## Description

BACKGROUND OF THE INVENTION

- 5 [0001] Multi-layers of  $\kappa$ - $\text{Al}_2\text{O}_3$  and  $\text{Ti}(\text{C},\text{O})$  or  $\kappa$ - $\text{Al}_2\text{O}_3$  and  $\text{TiN}$  have proved to exhibit better wear properties than single oxide layers, see U.S. Patent 5,700,569 and U.S. Serial No. 09/717,006.
- [0002] The deposition process of these prior art multi-layers is, however, relatively long and deposition is usually carried out at relatively high temperatures (usually at about  $1000^\circ\text{C}$ ), resulting in the transformation of kappa-alumina to alpha-alumina. The volume shrinkage encountered in the phase transformation will reduce adhesion of the alumina layers. As a result, adhesion problems in production will occur.
- 10 [0003] It has usually been thought that deposition temperatures of about  $1000^\circ\text{C}$  or higher are needed to deposit  $\text{Al}_2\text{O}_3$  coatings. As shown in the recent U.S. Application Serial No. 09/498,344,  $\text{Al}_2\text{O}_3$  can be deposited at the deposition temperatures about or exceeding  $800^\circ\text{C}$ , but less than  $1000^\circ\text{C}$ . Further, it was shown that the two  $\text{Al}_2\text{O}_3$  phases,  $\kappa$  and  $\gamma$ , could be deposited in a controlled way.
- 15 [0004] It has recently been confirmed that  $\text{Ti}(\text{C},\text{N})$  exhibits better wear resistance against crater wear and flank wear in hypoeutectoid steels than  $\text{TiN}$  (U.S. Serial No. 09/207,687). In recent in-house cutting tests, it has also been found that in hypereutectoid steel,  $\text{Ti}(\text{C},\text{N})$  is better than  $\text{TiN}$ , especially with respect to flank wear. In hyper-eutectic steel,  $\text{Al}_2\text{O}_3$  is a superior coating material against crater wear. In recent cutting tests in-house, it has also been found that the adhesion of both  $\kappa$  and  $\gamma$  phases to the MTCVD  $\text{Ti}(\text{C},\text{N})$  deposited at  $800^\circ\text{C}$  is surprisingly good. By depositing  $\kappa$  or  $\gamma$  with MTCVD  $\text{Ti}(\text{C},\text{N})$  as a multi-layer, the wear properties of the prior art  $\text{TiN}/\text{Ti}(\text{C},\text{O})$ - $\kappa$  multi-layers could thus be enhanced.
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OBJECTS AND SUMMARY OF THE INVENTION

- 25 [0005] It is an object of this invention to avoid or alleviate the problems of the prior art.
- [0006] It is further an object of this invention to provide enhanced wear properties of  $\text{TiN}/\text{Ti}(\text{C},\text{O})$ - $\kappa$  multi-layers by depositing  $\kappa$  or  $\gamma$  with MTCVD  $\text{Ti}(\text{C},\text{N})$  as a multi-layer.
- [0007] In one aspect of the invention there is provided a coated body wherein the coating comprises a multi-layer of  $\gamma$ - $\text{Al}_2\text{O}_3$ .
- 30 [0008] In another aspect of the invention there is provided a coated body wherein the coating comprises a multi-layer of  $\kappa$ - $\text{Al}_2\text{O}_3$  and/or  $\gamma$ - $\text{Al}_2\text{O}_3$  layers interspersed with layers of  $\text{Ti}(\text{C},\text{N})$  on a layer of  $\text{Ti}(\text{C},\text{N})$ .
- [0009] In another aspect of the invention there is provided a coated body wherein the coating comprises a multi-layer of  $\kappa$ - $\text{Al}_2\text{O}_3$  and  $\gamma$ - $\text{Al}_2\text{O}_3$ , each applied by a chemical vapor deposition at a temperature of from  $700$  to  $900^\circ\text{C}$ .
- [0010] In another aspect of the invention there is provided a coated body wherein the coating comprises a multi-layer of  $\kappa$ - $\text{Al}_2\text{O}_3$  and/or  $\gamma$ - $\text{Al}_2\text{O}_3$  layers interspersed with layers of  $\text{Ti}(\text{C},\text{N})$  on a layer of  $\text{Ti}(\text{C},\text{N})$ .
- 35 [0011] In yet another aspect of the invention there is provided a coated body wherein the coating comprises a multi-layer of  $\kappa$ - $\text{Al}_2\text{O}_3$  and/or  $\gamma$ - $\text{Al}_2\text{O}_3$  layers interspersed with layers of  $\text{Ti}(\text{C},\text{N})$  on a layer of  $\text{Ti}(\text{C},\text{N})$  and with a layer of  $\text{Ti}(\text{C},\text{N})$  atop of the said multi-layer.

40 BRIEF DESCRIPTION OF THE DRAWINGS

- [0012] Fig. 1 is a representation of a coated body of the present invention including  $\kappa$ - $\text{Al}_2\text{O}_3$  multi-layers (Fig. 1a),  $\gamma$ - $\text{Al}_2\text{O}_3$  with multi-layers (Fig. 1b) and a mixed  $\gamma$  and  $\kappa$ - $\text{Al}_2\text{O}_3$  multi-layer (Fig. 1c).
- 45 [0013] Fig. 2 is a representation of a coated body of the present invention useful in cutting of SS1672 (Fig. 2a) and SS2258 (Fig. 2b).
- [0014] Fig. 3 shows Scanning Electron Microscope (SEM) images of the cutting edges of single and multi-layer  $\kappa$ - $\text{Al}_2\text{O}_3$  coated inserts after turning of 2, 5 and 8 minutes (9 minutes for the multi-layer  $\kappa$ - $\text{Al}_2\text{O}_3$  coated insert) of SS2258.
- [0015] Fig. 4 shows Scanning Electron Microscope (SEM) images of the cutting edges of single and multi-layer  $\kappa$ - $\text{Al}_2\text{O}_3$  coated inserts after turning of 2, 9 and 15 minutes of SS1678.
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DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS OF THE INVENTION

- [0016] Fig. 1 shows a schematic of the coating layer according to this invention. The coating is composed of the following layers:
- 55 [0017] 1. MTCVD  $\text{Ti}(\text{C},\text{N})$  base layer, deposition temperature  $800$ - $900^\circ\text{C}$
- [0018] 2. Multi-layer structure consisting of  $\kappa$ - $\text{Al}_2\text{O}_3$  (Fig. 1a),  $\gamma$ - $\text{Al}_2\text{O}_3$  (Fig. 1b) or both (Fig. 1c), together with MTCVD  $\text{Ti}(\text{C},\text{N})$  interlayers, all deposited at a temperature of  $800$ - $900^\circ\text{C}$ . The wear properties of  $\gamma$  are not yet fully elucidated. However,  $\gamma$  is a less stable phase than  $\kappa$  and can be used only in the uppermost layers of the multi-coating structure

which will be subjected to the shortest annealing during deposition.

[0019] The  $\text{Al}_2\text{O}_3$  layers in the multi-layer (whether  $\gamma$  or  $\kappa$ ) have an individual thickness of from about 0.1 to 3.2 microns, preferably about 0.3 to 1.2 microns. The  $\text{Ti}(\text{C},\text{N})$  layers in the multi-layer have an individual thickness of from about 0.1 to about 3.2 microns, preferably from about 0.3 to about 1.2 microns. The total thickness of the multi-layer is from about 3 to about 30 microns, preferably from about 5 to 15 microns. The multi-layer may also be deposited onto a  $\text{Ti}(\text{C},\text{N})$  layer of from about 2 to about 15 microns, preferably from about 3 to 10 microns, which in this instance is the first layer applied onto the body. In addition, a  $\text{Ti}(\text{C},\text{N})$  layer of the same thickness can be deposited atop the outermost layer of the alumina.

[0020] The body is preferably formed of a cemented carbide, a cermet, a ceramic, or a high speed steel and the coated body is preferably used as a cutting tool in metal cutting operations.

[0021] The  $\gamma$ - and  $\kappa$ - $\text{Al}_2\text{O}_3$  layers as well as the  $\text{Ti}(\text{C},\text{N})$  layers are applied by MTCVD (Medium Temperature Chemical Vapor Deposition). The deposition of the  $\gamma$ - and  $\kappa$ - $\text{Al}_2\text{O}_3$  layers utilize the technique described in my copending U.S. Patent Application Serial No. 09/498,334, herein incorporated by reference. In that process,  $\text{H}_2\text{S}$  is added to the otherwise conventional MTCVD techniques and apparatus in amounts greater than 0.7 vol %, generally 0.75 to 1.7 vol %, preferably greater than 1 up to about 1.5 vol %, of the total gaseous mixture.

[0022] The coating process is performed at temperatures of from about 700 to 900°C, preferably 750 to 850°C, at a pressure of from about 50 to 600 mbar, preferably from about 100 to 300 mbar, for a time sufficient to form the coating, generally from about 2 to 10 hours, preferably from about 4 to 8 hours.

[0023] It should be noted that the deposition of  $\text{Al}_2\text{O}_3$  can be carried out at the same temperature as the MTCVD  $\text{Ti}(\text{C},\text{N})$  layers, resulting in considerably shorter processes (the heating up/cooling down steps are eliminated) and the deposition of the multi-layer is carried out at relatively low temperature, resulting in no phase transformations. As a result, enhanced adhesion will be obtained and production yield will be enhanced. The multi-layer coating can be composed of both  $\kappa$  and  $\gamma$  which can simply be controlled by  $\text{H}_2\text{S}$ . The  $\gamma$  phase which is less stable than  $\kappa$  should be situated in the uppermost part (i.e., top half) of the multi-coating layer, if used; and as shown earlier, MTCVD  $\text{Ti}(\text{C},\text{N})$  exhibits better wear resistance than  $\text{TiN}$ . By using  $\text{Ti}(\text{C},\text{N})$  instead of  $\text{TiN}$ , crater wear resistance in hypo-eutectic steels and flank wear resistance in hyper-eutectic steels will be enhanced.

[0024] The invention is additionally illustrated in connection with the following Examples which are to be considered as illustrative of the present invention. It should be understood, however, that the invention is not limited to the specific details of the Examples.

#### EXAMPLE 1

[0025] Tables 1 and 2 show a summary of the wear properties of different coating in SS1672 and SS2258 (hypo- and hyper-eutectic steels, respectively)

Table 1 -

SS1672				
	Crater Wear	Flank Wear	Notch Wear	Deformation
$\alpha$ - $\text{Al}_2\text{O}_3$	---	---	++	++
$\kappa$ - $\text{Al}_2\text{O}_3$	---	---	++	+++
TiCN(MTCVD)	++	+++	-	-
TiCN (CVD)	+++	+++	-	-
TiN (CVD)	+	++	++	--
TiC (CVD)	+	+++	-	-

Table 2 -

SS2258				
	Crater Wear	Flank Wear	Notch Wear	Deformation
$\alpha$ - $\text{Al}_2\text{O}_3$	+++	--	++	++
$\kappa$ - $\text{Al}_2\text{O}_3$	+++	-	++	+++
TiCN(MTCVD)	--	+++	-	-

Table 2 - (continued)

SS2258				
	Crater Wear	Flank Wear	Notch Wear	Deformation
TiCN (CVD)	--	+++	-	-
TiN (CVD)	--	+	-	--
TiC (CVD)	---	+++	-	-

[0026] As can be seen, the coating material may show very different behaviors in these steels. Consequently, different coating structures have to be developed for SS1672 (thin  $\text{Al}_2\text{O}_3$  layers + thick  $\text{Ti}(\text{C},\text{N})$  layers, Fig. 2a) and for SS2258 (thick  $\text{Al}_2\text{O}_3$  layers + thin  $\text{Ti}(\text{C},\text{N})$  layers, Fig. 2b) in accordance with the knowledge of the skilled artisan. Schematics of the optimized coating structures for these steel are shown in Figs. 2a and 2b.

#### EXAMPLE 2

[0027] Cutting tests were performed in SS1672 and SS2258. The coating, according to Fig. 2a, being composed of 6  $\kappa\text{-Al}_2\text{O}_3$  layers interspersed by layers of  $\text{Ti}(\text{C},\text{N})$ , (total multi-layer thickness 7  $\mu\text{m}$ ) was tested on SS1672 and a coating deposited according to Fig. 2b, being composed of 5  $\kappa\text{-Al}_2\text{O}_3$  layers interspersed by layers of  $\text{Ti}(\text{C},\text{N})$  (total multi-layer thickness 7  $\mu\text{m}$ ) was tested on SS2258. Cutting tests were also conducted using inserts having single layers of  $\text{Al}_2\text{O}_3$ , TiN and  $\text{Ti}(\text{C}, \text{N})$  as well as a multi-layer of  $\kappa$ -alumina and TiN were compared. The results are given in Tables 3 and 4.

Table 3.-

SS1672, Cutting Speed 250 m/min		
Coating	Life time/min	Failure Mode
$\text{Al}_2\text{O}_3$	11	crater wear
TiN	15	crater wear + notch
TiCN	19	crater wear + notch
Multi $\kappa$ + TiN	25	crater wear
Multi $\kappa$ + TiCN	39	crater wear

Table 4 -

SS2258, Cutting Speed 200 m/min		
Coating	Life time/min	Failure Mode
$\text{Al}_2\text{O}_3$	15	flank wear
TiN	8	crater wear
TiCN	8	crater wear
Multi $\kappa$ + TiN	25	flank wear
Multi $\kappa$ + TiCN	32	flank wear

#### EXAMPLE 3

[0028] A detailed comparison of the behaviors of a single layer and a multi-layer coating (Fig. 2b) in turning SS2258, is presented in Fig. 3. The multi-layer coating is superior to the single layer SS2258. In this steel, the flank wear is clearly reduced by the  $\text{Ti}(\text{C},\text{N})$  coatings. It is clear from the SEM micrograph that both crater wear and flank wear resistance of the multi-layer  $\kappa\text{-Al}_2\text{O}_3$  coated inserts were superior to those of the single  $\kappa\text{-Al}_2\text{O}_3$  coated inserts. In this kind of steel where alumina-coated inserts in general perform well, the effects of the multi-layering is very clear. The lifetime of the insert is drastically increased, in particular at 200 m/min more than about 100%. In ball-bearing steel, SS2258 (hypereutectoid steel), a multi layering results in much more drastically reduced wear than observed earlier

in hypoeutectoid steels (U.S. Patent 5,700,569).

#### EXAMPLE 4

[0029] A detailed comparison of the behaviors of a single layer and a multi-layer coating (Fig. 2b) in turning of SS1672, is presented in Fig. 4. The multi-layer coating is superior to the single layer also in SS1672. Compared with multilayer  $\kappa$ - $\text{Al}_2\text{O}_3$  coatings according to U.S. Patent 5,700,569, multilayers of  $\kappa$ - $\text{Al}_2\text{O}_3$  with Ti(C,N) together with reduced thickness of the alumina layers enhanced the performance of the inserts over the prior art. It appears clear from the SEM micrograph that both crater wear and flank wear resistances were superior to those exhibited by the single layer. In this steel, the flank wear is clearly reduced more than earlier observed when multi-layer coatings of  $\kappa$ - $\text{Al}_2\text{O}_3$  and Ti(C, O) were investigated (U.S. Patent 5,700,569).

[0030] The principles, preferred embodiments and modes of operation of the present invention have been described in the foregoing specification. The invention which is intended to be protected herein, however, is not to be construed as limited to the particular forms disclosed, since these are to be regarded as illustrative rather than restrictive. Variations and changes may be made by those skilled in the art without departing from the spirit of the invention.

#### Claims

1. A coated body wherein the coating comprises a multi-layer of  $\gamma$ - $\text{Al}_2\text{O}_3$ .
2. A coated body wherein the coating comprises a multi-layer of  $\kappa$ - $\text{Al}_2\text{O}_3$  applied by a chemical vapor deposition at a temperature of from 700 to 900°C.
3. A coated body wherein the coating comprises a multi-layer of  $\kappa$ - $\text{Al}_2\text{O}_3$  and  $\gamma$ - $\text{Al}_2\text{O}_3$ , each applied by a chemical vapor deposition at a temperature of from 700 to 900°C.
4. The coated body of any of claims 1, 2 and 3 wherein the said multi-layers of  $\gamma$ - and/or  $\kappa$ - $\text{Al}_2\text{O}_3$  are interspersed with layers of Ti(C,N).
5. The coated body of claim 4 wherein the multi-layers of  $\gamma$  and/or  $\kappa$ - $\text{Al}_2\text{O}_3$  comprise layers having a thickness of from about 0.1 to about 3.2 microns.
6. The coated body of claim 4 wherein the multi-layers of  $\gamma$  and/or  $\kappa$ - $\text{Al}_2\text{O}_3$  comprise layers having a thickness of from about 0.3 to about 1.2 microns.
7. The coated body of claim 4 wherein the layers of Ti(C,N) comprise layers having a thickness of from about 0.1 to about 3.2 microns.
8. The coated body of claim 4 wherein the layers of Ti(C,N) comprise layers having a thickness of from about 0.3 to about 1.2 microns.
9. The coated body of any of claims 1, 2 and 3 wherein a layer of Ti(C,N) is atop the said multi-layers of  $\gamma$  and/or  $\kappa$ - $\text{Al}_2\text{O}_3$ .
10. The coated body of claim 3 wherein the  $\gamma$ - $\text{Al}_2\text{O}_3$  layers comprise the uppermost alumina layers and the  $\kappa$ - $\text{Al}_2\text{O}_3$  layers comprise the innermost alumina layers.
11. The coated body of any of claims 1, 2, and 3 wherein the said multi-layers have a total thickness of from about 3 to about 30 microns.
12. The coated body of claim 11 wherein the said multi-layers have a total thickness of from about 5 to about 15 microns.
13. The coated body of any of claims 1, 2, and 3 wherein the said body is selected from the group consisting of a cemented carbide, cermet, ceramic, high speed steel and mixtures thereof.
14. A metal cutting tool made from the coated body of claim 13.

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15. A coated body wherein the coating comprises a multi-layer of  $\kappa\text{-Al}_2\text{O}_3$  and/or  $\gamma\text{-Al}_2\text{O}_3$  layers interspersed with layers of  $\text{Ti(C,N)}$  on a layer of  $\text{Ti(C,N)}$ .

5 16. A coated body wherein the coated comprises a multi-layer of  $\kappa\text{-Al}_2\text{O}_3$  and/or  $\gamma\text{-Al}_2\text{O}_3$  layers interspersed with layers of  $\text{Ti(C,N)}$  on a layer of  $\text{Ti(C,N)}$  and with a layer of  $\text{Ti(C,N)}$  atop of the said multi-layer.

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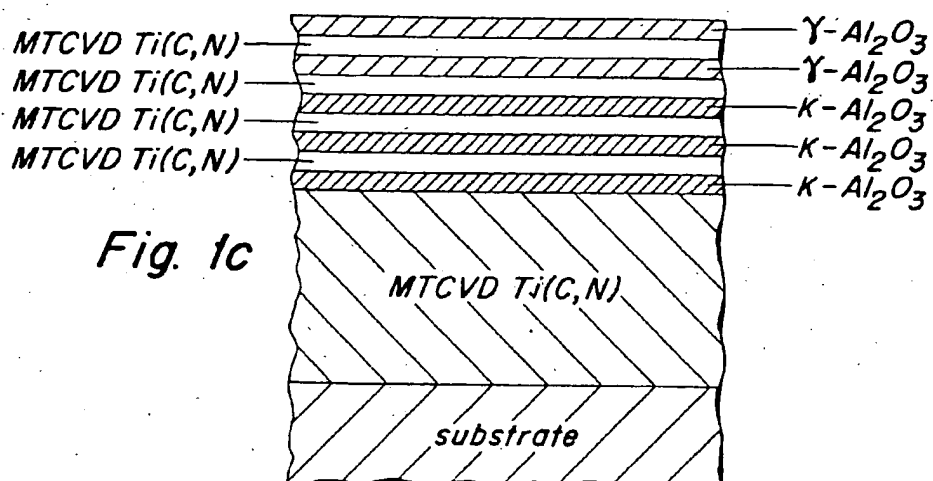
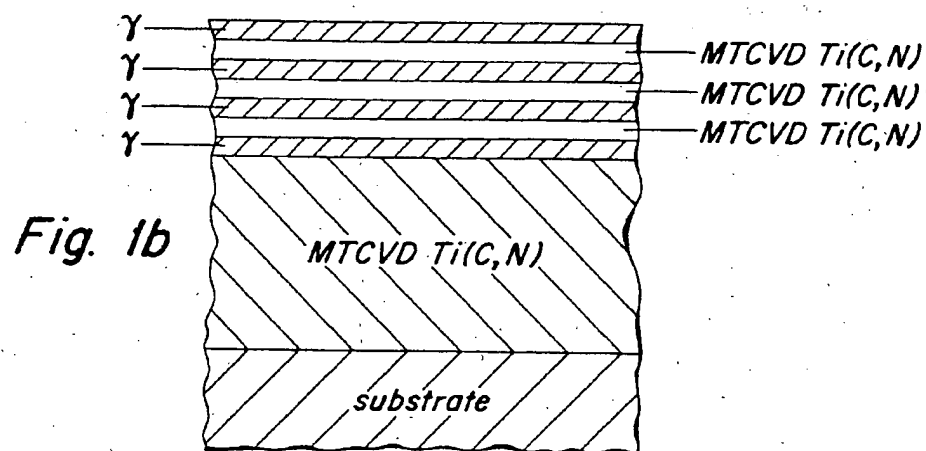
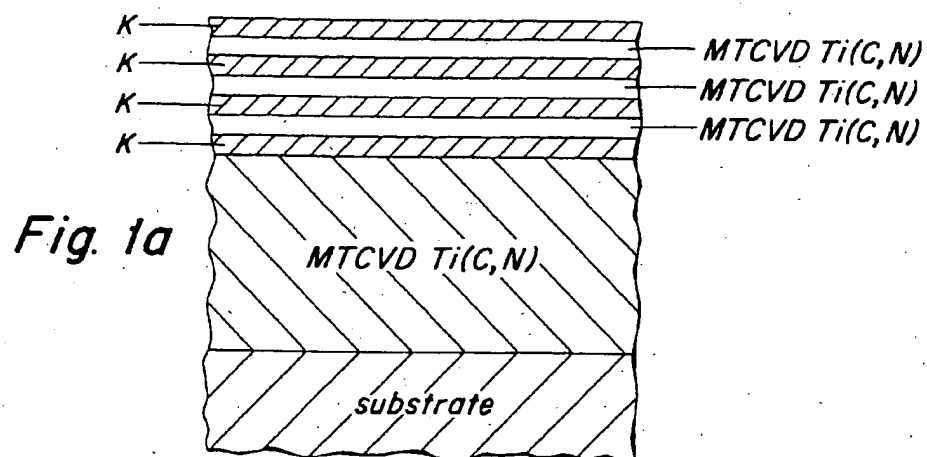




Fig. 2a

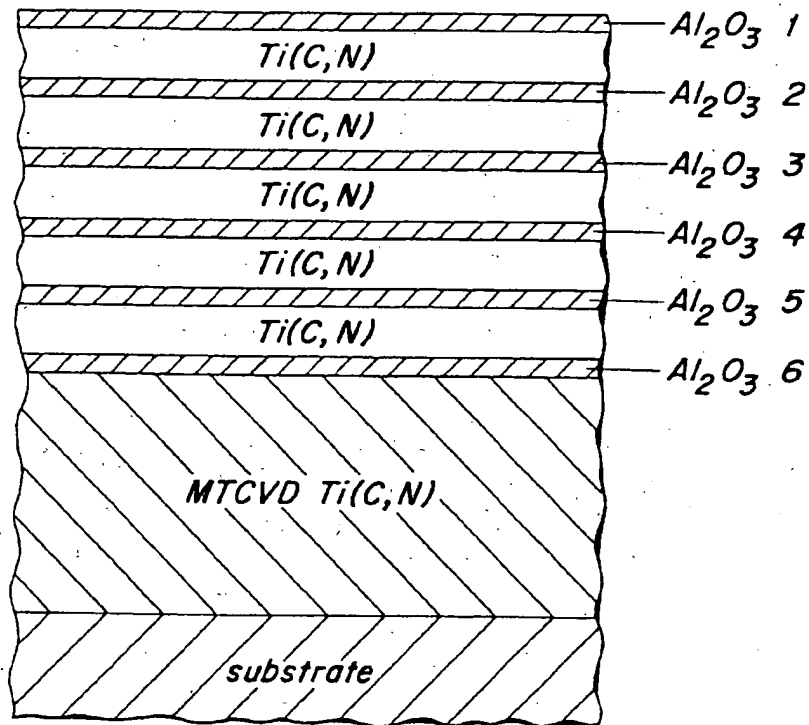
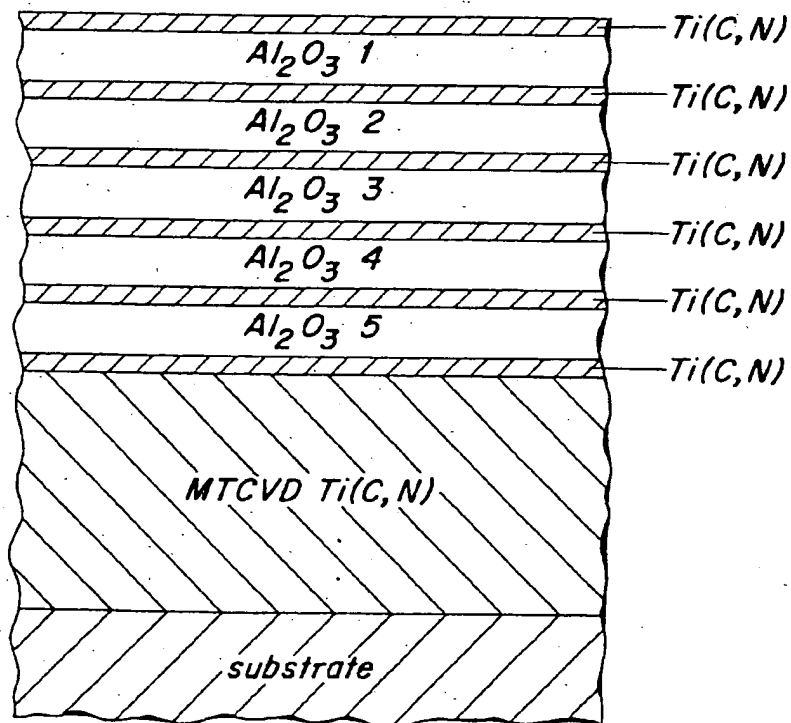
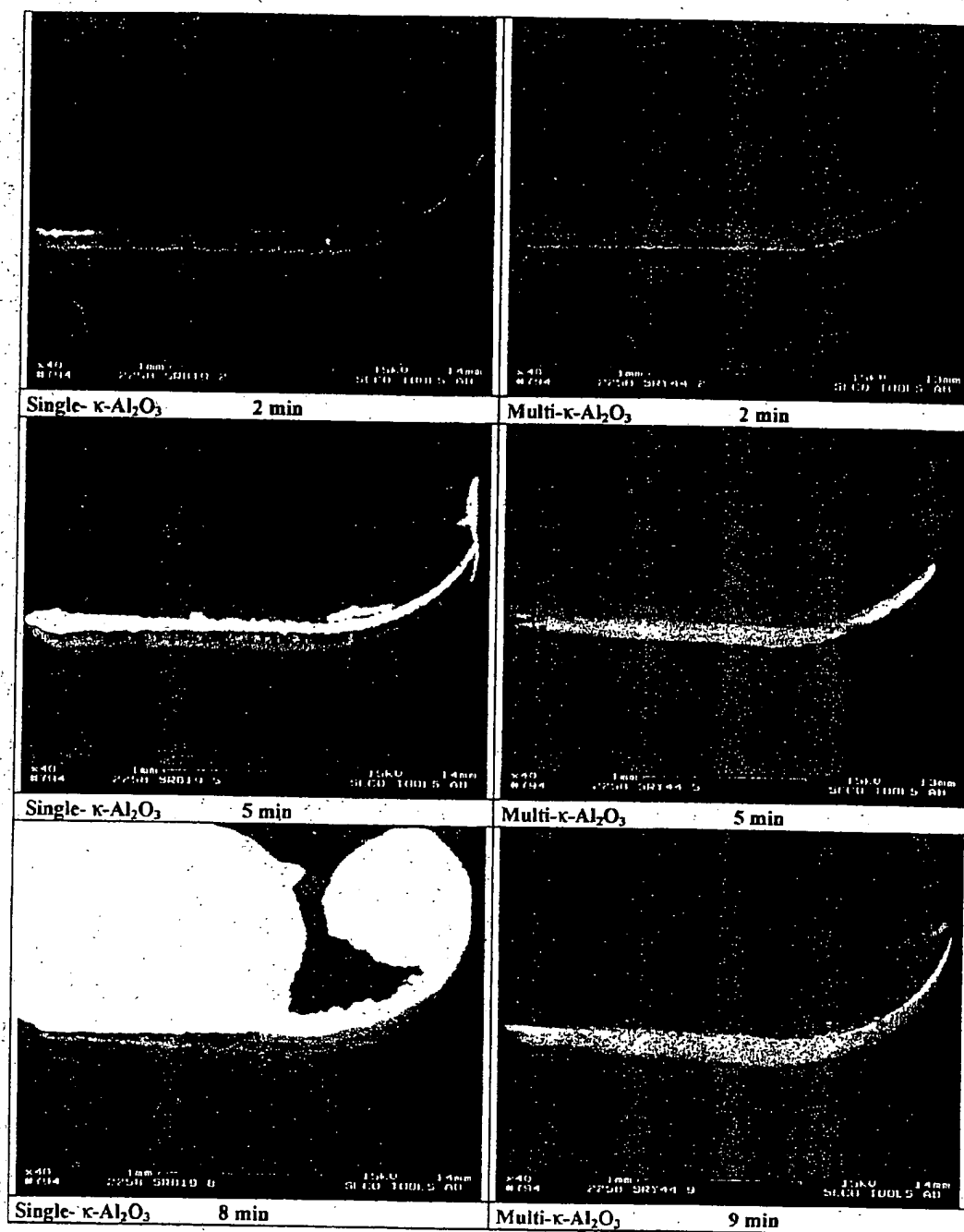


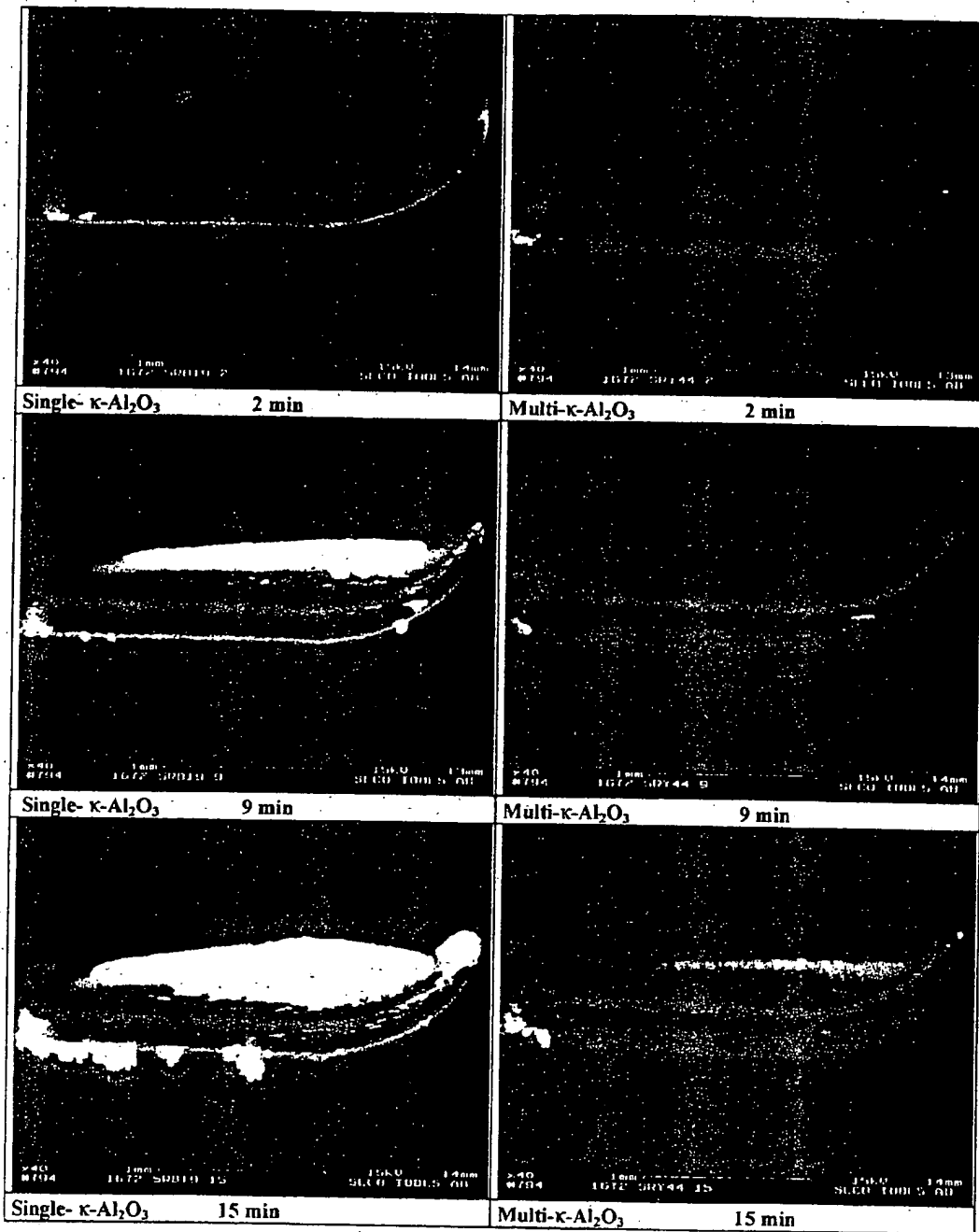
Fig. 2b





SS 2258  
 $v_c = 275\text{m/min}$   
 $f = 0.4\text{ mm/r}$   
 $a_p = 2.5\text{ mm}$

Fig. 3



SS 1672  
 $v_c = 250\text{m/min}$  , cutting time 2, 9 and 15 min  
 $f = 0.4\text{ mm/r}$   
 $a_p = 2.5\text{ mm}$

Fig. 4



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## EUROPEAN SEARCH REPORT

Application Number  
EP 02 44 5036

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Place of search <b>MUNICH</b>		Date of completion of the search <b>18 June 2002</b>	Examiner <b>Brisson, O</b>
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